



MANUFACTURING PROCESS FOR A HIGH STRENGTH WORK HARDENED
PRODUCT MADE OF AlZnMgCu ALLOY

Background of the invention

Field of the invention

The invention relates to the manufacture of work-hardened products by rolling, extrusion or forging made of a high mechanical strength aluminium alloy of the AlZnMgCu type, particularly products used for aeronautical construction and particularly for upper wing members of aircrafts.

Background art

Al-Zn-Mg-Cu type alloys have been used for aircraft construction for more than 50 years, and particularly for upper wing members. Thus, 7075, 7178, 7050, 7150 alloys and more recently 7055 and 7449 alloys have been used. These alloys have usually been used in the T6 temper, in other words annealing corresponding to the maximum tensile yield strength, or an over annealed temper T76, T79 or T77 to obtain better corrosion resistance. As an illustration of this state of the art, there are patents EP 0020505 by Boeing related to the 7150 alloy, patents US 4,477,292, US 4,832,758, US 4,863,528 and US 5,108,520 by Alcoa on the T77 treatment, Alcoa' patent EP 0377779 dealing with a manufacturing process for the 7055 alloy, and the applicant's patent application EP 0670377 describing a process for the manufacture of plates made of 7449 alloy.

The properties of the 7449 alloy developed by the applicant for plates intended for use on upper wing

members have been studied in the paper written by T. Warner et al. "Aluminium alloy developments for affordable airframe structures", Conference on Synthesis, Processing and Modelling of Advanced Materials, ASM International, Paris, June 25-27 1977, pp 77-88. Figure 2 in the article reproduced as figure 1 in this application, shows typical properties of plates from 15 to 40 mm thick made of this alloy, specifically the ultimate strength and the tensile yield strength in the L direction, the compression yield strength in the L direction and the stress corrosion limit (in the ST direction), in the T651 temper and in a T7x51 temper with improved corrosion resistance. This temper was identified in later publications by the same authors as T7951 (or T79511 for extruded products), for example in the paper written by F. Heymès et al. "New aluminium semi-products for airframe application", METEC congress, Düsseldorf, June 1999 that uses the same figure. Figure 1 attached to this application shows that the compression yield strength in the T79 temper is lower than the corresponding yield strength in the T6 temper. In the T7951 temper, plates made of 7449 have a 10% higher compression yield strength, better resistance to exfoliation corrosion and to stress corrosion and fatigue than plates made of 7150 in the T651 temper that are usually used for upper wing members of commercial aircrafts, without any reduction in the tolerance to damage.

In summary, the state of the art shows firstly that the mechanical compression strength is an essential property for upper wing members, and also that manufacturers of high strength alloys offer

products for this application either in the T6 temper corresponding to the maximum tensile yield strength, or an over annealed T7 temper with better corrosion resistance but with lower mechanical strength.

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Brief description of the invention

The purpose of the invention is to further improve the mechanical compression strength of products made of high strength 7000 alloys intended particularly for upper wing members of aircrafts without losing any other usage properties.

The purpose of the invention is a process for manufacturing a work-hardened product made of a high mechanical strength Al-Zn-Mg-Cu aluminium alloy comprising:

- casting an ingot made of an alloy with composition (% by weight) Zn (7.0 - 11.0), Mg (1.8 - 3.0), Cu (1.2 - 2.6), at least one of the elements Mn (0.05 - 0.4), Cr (0.05 - 0.30), Zr (0.05 - 0.20), Hf (0.05 - 0.5), V (0.05 - 0.3), Ti (0.01 - 0.2) and Sc (0.05 - 0.3), the remainder being made of aluminium and inevitable impurities,
- possibly homogenisation of said ingot,
- hot transformation of said ingot by rolling, extrusion or forging,
- solution heat treatment and quenching of the product obtained,
- possibly controlled stretching with a permanent set between 1 and 5%,
- annealing of the product at a temperature and for a duration such that the product reaches the peak compression yield strength in the L direction.

Another purpose of the invention is the rolled, extruded or forged product obtained by said process.

Another purpose of the invention is the structural element for mechanical construction, and particularly
 5 for aeronautical construction, made from at least one rolled, extruded or forged product obtained according to said process.

Brief description of the figures

10 Figure 1 shows typical properties, namely the tensile yield strength (L direction), the ultimate tensile strength (L direction), the compression yield strength (L direction) and the stress corrosion threshold (ST direction) for plates between 15 and 40
 15 mm thick made of the 7150-T651, 7449-T651 and 7449-T7951 alloys according to prior art.

Figure 2 shows the annealing time-temperature domain in the process according to the invention.

Figure 3 shows the ultimate strength and the
 20 tensile yield strength of 38 mm thick plates made of 7449 alloy in example 1 as a function of equivalent annealing time at 120°C for different annealing temperatures.

Figure 4 shows the ultimate tensile strength (L
 25 direction) and the tensile and compression yield strength (L direction) of 38 mm plates made of 7449 alloy in example 2 as a function of the equivalent annealing time at 120°C.

Figure 5 shows the compression yield strength of
 30 plates made of alloys A and B in example 3 as a function of the equivalent annealing time at 120°C

Detailed description of the invention

The invention is based on demonstrating a shift between the peak mechanical tensile strength obtained with annealing, corresponding to what is usually called the T6 temper, and the peak mechanical compression strength. Although it has been known for a long time that the upper wing members are stressed mainly in compression, and that therefore the compression yield strength controls the dimensions of structural elements of this part of the wing, metallurgists have always used the tensile strength to define the T6 temper of maximum strength achieved in annealing.

The inventors have found that there is a metallurgical state between tempers T6 and T79 according to prior art, in which the compression yield strength passes through a peak of between 20 and 25 MPa above the compression yield strength values of these two tempers.

The process according to the invention is applicable to Al-Zn-Mg-Cu type alloys with a high zinc content between 7 and 11%, with a magnesium content between 1.8 and 3%, and preferably between 1.8 and 2.4%, and a copper content between 1.2 and 2.6% and preferably between 1.6 and 2.2%. The invention is not particularly useful for a zinc content below 7% since these types of alloy are no longer used in aeronautical construction for the fabrication of structural elements stressed in compression. For zinc contents higher than 11%, difficulties are encountered during industrial casting of rolling ingots or billets large enough for the production of plates, sections or forged parts suitable for the manufacture of the said structural elements.

The process according to the invention is applicable particularly for alloys used for manufacturing elements of the upper wing members of aircrafts, for example 7055, 7349 and 7449 alloys in the form of work hardened products, in other words rolled, extruded or forged products. This process comprises the manufacture of an ingot, namely a rolling ingot for rolled products, a billet or extrusion ingot for extruded products or a forging ingot for forged products, in a known manner. This ingot is preferably homogenised at a temperature close to the incipient melting temperature of the alloy, as described in patent application EP 0670377. It is then transformed by hot rolling, extrusion or forging, to the required dimension. The product obtained is solution heat treated also at a temperature fairly close to the incipient melting temperature of the alloy, this temperature being controlled by differential enthalpic analysis. Solution heat treatment is followed by quenching, usually in cold water. The quenched product is preferably subjected to controlled stretching with a permanent set of between 1 and 5%.

The product is then annealed to obtain the peak compression yield strength in the L direction. Annealing may be done in a single step, in other words include a temperature rise gradient that may or may not be linear as a function of time, a period of time at a constant temperature within the limit of the temperature tolerance of the furnace used, and cooling down to ambient temperature. For single step annealing, the constant temperature is between 120 and 150°C with a duration within the parallelogram AEFG in figure 2 and preferably between 120 and 145°C with a duration

within the parallelogram ABCD in figure 2. The latter annealing process is a particularly preferred embodiment of this invention. For example, it can be used for products made from the 7449 and 7439 alloys.

5 Annealing may also be done in two steps, with a first step at a temperature between 80 and 120°C, and a second step at a higher temperature between 120 and 160°C. It may also be done in three steps, with a first and a second step within the same limits as for the
10 two-step annealing, and a third step at a lower temperature than the second step, between 100 and 140°C. Considering the time necessary for temperature rises in industrial furnaces, it is difficult to envisage steps lasting less than 2h, and preferably
15 less than 5h.

In all cases, the two parameters (temperature and duration) can be converted to a single parameter, the equivalent time at 120°C defined by the following formula:

$$20 \quad T(\text{eq}) = \frac{\int \exp(-16000/T) dt}{\exp(-16000/T_{\text{ref}})}$$

where T is the temperature of the annealing step in °K, t is the treatment duration in hours and T_{ref} is the reference temperature in this case assumed to be 120°C, namely 393°K. The equivalent annealing time at
25 120°C is between 100 and 250 h, and 50 to 200 h more than the equivalent annealing time for T651 annealing. The annealing time necessary to reach the peak compression yield strength depends on the composition of the alloy and particularly the Cu/Mg ratio, the
30 necessary duration increasing with this ratio.

The work hardened product, and particularly the rolled, extruded or forged product obtained using the

process according to the invention, can advantageously be used for the manufacture of structural elements, particularly for aeronautical construction. Due to the increase in the compression yield strength resulting from the process according to the invention, a structural element manufactured from at least one extruded, rolled or forged product according to the invention has a better resistance to compression loads than a structural element with the same dimensions made from work hardened, extruded or forged products according to prior art. In one preferred embodiment of the invention, the structural element is an upper wing member of an aircraft.

15 Examples

Example 1

38 mm thick plates are made from 7449 alloy. The composition of the alloy is (% by weight) Zn = 8.11, Mg = 2.19, Cu = 1.94, Si = 0.04, Fe = 0.07, Zr = 0.09, Cr = 0.005, Ti = 0.025, the remainder aluminium and impurities (< 0.05 each).

Plates have been pre-widened to increase the plate width from 1100 mm to 2500 mm, hot rolled to 38 mm with an exit temperature of 378°C, solution heat treatment at 475°C, quenching with cold water and controlled stretching to 2.8% permanent set after waiting for 1 h after quenching.

Samples taken from the mid-thickness of the plates were subjected to 11 different single-step or two-step type annealings as shown in table 1. The rise and fall gradients between steps being 16°C/h and 65°C/h respectively, corresponding to the rates observed for

industrial heat treatment furnaces. For each annealing, the equivalent time at 120°C t_{eq} is calculated using the following formula:

$$t(eq) = \frac{\int \exp(-16000/T) dt}{\exp(-16000/T_{ref})}$$

- 5 where T is the temperature of the annealing step in °K, t is the treatment duration in hours and T_{ref} is the reference temperature, in this case assumed to be 120°C, namely 393°K.

10 The 11 tested annealings were between annealing T651 and annealing T7951 according to prior art, and their parameters and the corresponding equivalent times are shown in table 1.

15 In each case, the static tensile properties in the L direction were measured (ultimate tensile strength R_m , tensile yield strength $R_{0.2}$ and elongation A) on TOR 6 test pieces taken from the central part of the plates. The results are the average of at least two measurements and are shown in table 1 and in figure 3.

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Table 1

| Annealing 1 st step | Annealing 2 nd step | T_{eq} at 120°C (h) | $R_{0.2(L)}$ (MPa) | $R_{m(L)}$ (MPa) | A (%) |
|-----------------------------------|-----------------------------------|-----------------------------|-----------------------|---------------------|----------|
| 24 h- 120°C | | 24 | 617 | 661 | 12 |
| 48 h- 120°C | | 48 | 623 | 661 | 12 |
| 96 h- 120°C | | 96 | 624 | 655 | 12 |
| 6 h-135°C | | 29 | 616 | 655 | 12 |
| 12 h- 135°C | | 55 | 619 | 651 | 11 |
| 24 h- 135°C | | 108 | 619 | 651 | 11 |
| 48 h- 135°C | | 215 | 611 | 642 | 11 |
| 24 h- | 5 h-150°C | 125 | 620 | 649 | 11 |

| | | | | | |
|----------------|----------------|-----|-----|-----|----|
| 120°C | | | | | |
| 24 h- 120°C | 9 h-150°C | 196 | 613 | 642 | 11 |
| 24 h- 120°C | 13 h- 150°C | 265 | 607 | 636 | 10 |
| 24 h- 120°C | 17 h- 150°C | 336 | 595 | 627 | 10 |

It is found that close to the peak, the annealing processes with the lowest temperature, in other words 120°C, give the highest values of $R_{0.2}$ and R_m . For two-
5 step annealing processes, this effect is controlled by the temperature of the second step. Furthermore, the peaks for $R_{0.2}$ and R_m are similar, but not at exactly the same location. The T651 peak treatment can be
10 defined as being the treatment that results in values of $R_{0.2}$ and R_m within 5 MPa of the maximum potential value, while remaining industrially acceptable. In this case, it is a 48 h treatment at 120°C.

Example 2

Samples taken from 38 mm thick plates made of 7449 alloy with composition Zn = 8.38, Mg = 2.15, Cu = 1.96, Si = 0.04, Fe = 0.06, Zr = 0.11, the remainder aluminium and impurities (< 0.5% each) are made in exactly the same way as in example 1.

Eight different annealings were carried out on these samples, between annealing T651 defined in example 1 and annealing T7951. The temperatures and durations of these eight annealings and the corresponding equivalent times at 120°C are shown in table 2.

Table 2

| Annealing | Parameters | Equivalent time |
|-----------|-------------------------|-----------------|
| A (T651) | 48 h-120°C | 48 |
| B | 12 h-135°C | 48 |
| C | 18 h-135°C | 78 |
| D | 24 h-135°C | 102 |
| E | 30 h-135°C | 130 |
| F | 24 h-120°C +5.5 h-150°C | 130 |
| G | 24 h-120°C +11 h-150°C | 222 |
| H (T7951) | 24 h-120°C +17 h-150°C | 321 |

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In addition to the mechanical tensile properties, the compression yield strength was measured in the L direction on 13 mm diameter and 25 mm long test pieces, and the electrical conductivity was measured on samples taken from the surface. The average results of the two measurements are shown in table 3 and in figure 4, for R_m and $R_{0.2}$ in tension, and $R_{0.2}$ in compression.

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Table 3

| Annealing | R _m ten (MPa) | R _{0.2} ten (MPa) | A (%) | R _{0.2} comp (MPa) | Conduct (MS/m) |
|-----------|-----------------------------|-------------------------------|----------|--------------------------------|-------------------|
| A | 638 | 676 | 12.4 | 596 | 18.4 |
| B | 639 | 673 | 11.8 | 599 | 18.7 |
| C | 637 | 668 | 12.0 | 611 | 19.1 |
| D | 634 | 663 | 11.0 | 614 | 19.7 |
| E | 633 | 663 | 10.5 | 615 | 20.0 |
| F | 635 | 662 | 11.2 | 613 | 20.1 |
| G | 619 | 648 | 10.5 | 608 | 21.2 |
| H | 597 | 621 | 10.7 | 590 | 21.9 |

It is seen that the annealing that gives the peak compression yield strength (L direction) is at an equivalent time of the order of 150h, in other words at an equivalent time intermediate between T651 annealing and T7951 annealing. The useful range is between 100 and 250h of equivalent time at 120°C, which is 50 to 200h more than for a T651 annealing. This annealing that results in the compression peak gives an improvement of 19 MPa compared with a T651 annealing and 25 MPa compared with a T7951 annealing.

Example 3

Plates made of two 7449 alloys with the thicknesses and compositions indicated in table 4, were made in the same way as in the previous examples as far as quenching.

Table 4

| Alloy | e | Si | Fe | Cu | Mg | Zn | Zr | Ti |
|-------|---|----|----|----|----|----|----|----|
|-------|---|----|----|----|----|----|----|----|

| | (mm) | | | | | | | |
|---|------|-------|-------|------|------|------|------|------|
| A | 30 | 0.049 | 0.075 | 1.87 | 2.35 | 8.38 | 0.11 | 0.03 |
| B | 23 | 0.045 | 0.068 | 1.95 | 2.27 | 8.31 | 0.10 | |

These plates were annealed as described in table 5, the first 11 annealings being for alloy A and the last 7 for alloy B. The compression yield strength $R_{0.2}$ in the L direction, and the modulus of elasticity in compression also in the L direction, were measured on 13 mm diameter and 25 mm long test pieces taken from the central part of the plates. The results are shown in table 5, and the yield strength results are shown in figure 5 as a function of the equivalent annealing time at 120°C.

Table 5

| Annealing 1 st step | Annealing 2 nd step | Annealing 3 rd step | $R_{0.2}$ comp (MPa) | Modulus (MPa) |
|-----------------------------------|-----------------------------------|-----------------------------------|-------------------------|------------------|
| 24 h-80°C | 24 h-135°C | | 605 | 70281 |
| 24 h-100°C | 24 h-135°C | | 602 | 71200 |
| 24 h-120°C | 24 h-135°C | | 607 | 72335 |
| 24 h-100°C | 18 h-140°C | | 603 | 70598 |
| 24 h-100°C | 7 h-150°C | | 601 | 70618 |
| 24 h-100°C | 2.5 h-160°C | | 607 | 72302 |
| 24 h-100°C | 30 h-140°C | | 600 | 72806 |
| 24 h-100°C | 18 h-140°C | 24h-120°C | 616 | 71621 |
| 24 h-100°C | 7 h-150°C | 24h-120°C | 615 | 70862 |
| 24 h-100°C | 2.5 h-160°C | 24h-120°C | 622 | 72569 |
| T7951 | | | 587 | |
| 24 h-80°C | 24 h-135°C | | 635 | 72910 |
| 24 h- | 24 h-135°C | | 611 | 72222 |

| | | | | |
|----------------|------------|------------|-----|-------|
| 120°C | | | | |
| 24 h- 100°C | 18 h-140°C | | 614 | 73244 |
| 24 h- 100°C | 7 h-150°C | | 610 | 72349 |
| 24 h- 100°C | 30 h-140°C | | 596 | 70181 |
| 24 h- 100°C | 7 h-150°C | 24 h-120°C | 621 | 71303 |
| T7951 | | | 598 | |

5 It is found that the peak compression yield strength occurs for an equivalent annealing time at 120°C between 100 and 200 h, and that three-step annealing processes give higher values. Furthermore, it is found that the compression yield strength is about 15 MPa better than for the T7951 annealing for two-step annealing processes, and about 25 MPa better for three-step annealing processes.